
(12) UK Patent Application (19) GB (11) 2 103 593 A

(21) Application No 8124422

(22) Date of filing 10 Aug 1981

(43) Application published
23 Feb 1983

(51) INT CL³
B01D 53/00

(52) Domestic classification
C1A S181 S183 S186
S18Y S23X S410 S415
S416 S418 S41Y S471
S491 S492 S493 S601
S602 S60X S60Y S611
S612 S645 S681 S682
S711 SA SB
B1L 102 EA
U1S 1272 1484 B1L C1A

(56) Documents cited
GB A 2051761
GB 1417528
GB 1413836
GB 1306240
GB 0882536

(58) Field of search
B1L
C1A

(71) Applicants
Hunting Hivolt Limited,
(Great Britain),
Marlborough Road,
Churchill Industrial
Estate,
Lancing,
West Sussex,
BN15 8TU.

(72) Inventors
Roy Venton-Walters

(74) Agents
Lloyd Wise, Tregear and
Co.,
Norman House,
105-109 Strand,
London WC2R 0AE.

(54) Removal of hydrocarbon impurity
from O₂-containing gas stream

(57) An oxygen-containing gas stream, such as air, which contains hydrocarbon impurities and possibly also carbon monoxide, derived from example from passage through a compressor, is purified by bringing the gas stream into contact with an oxidation catalyst under conditions which convert the hydrocarbon contaminants into carbon dioxide and water, and thereafter the gas stream is brought into contact with a chemical purification agent, for example of the absorbent or chemical conversion type, to remove one or more contaminants present in the gas stream.

GB 2 103 593 A

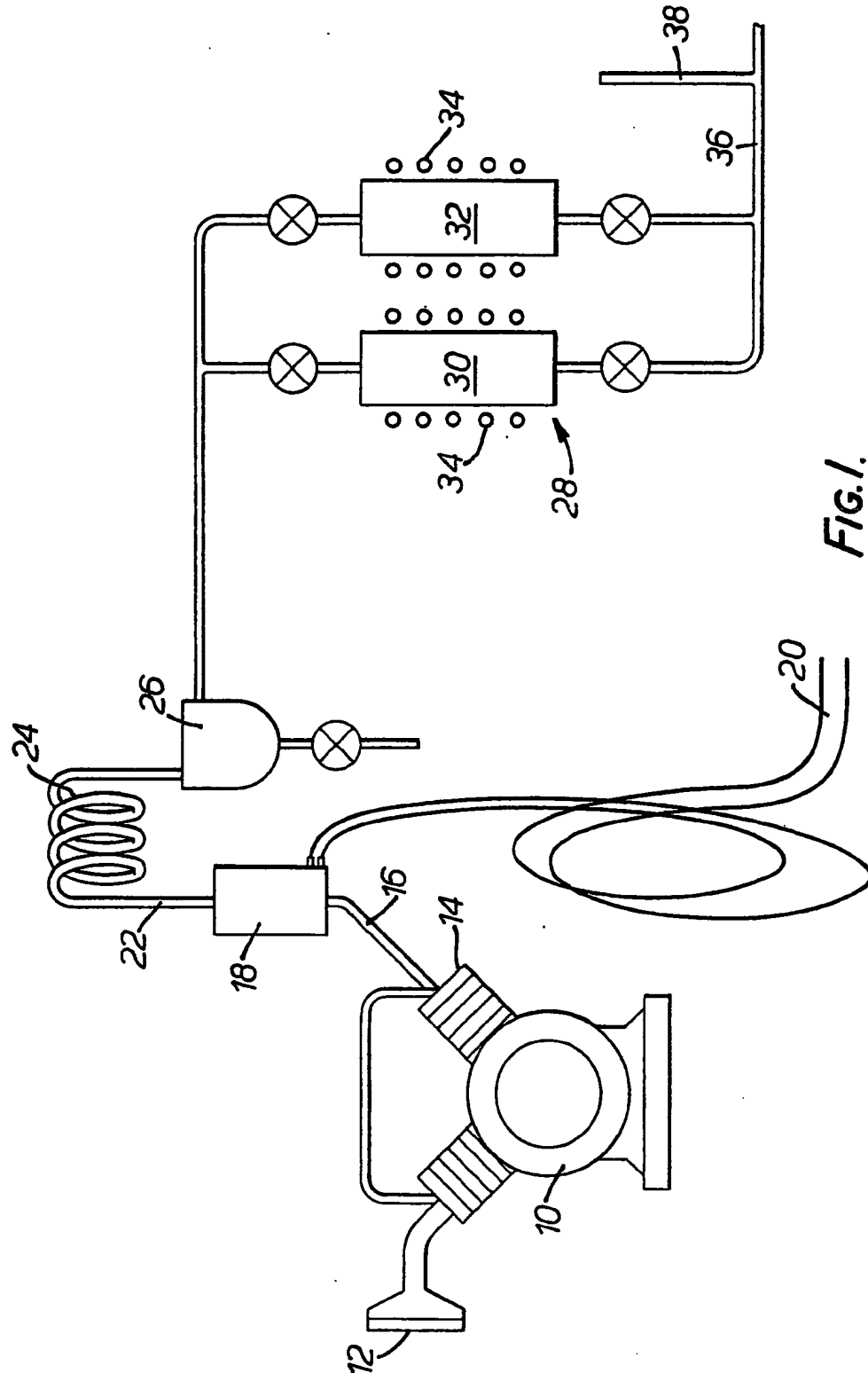


FIG. 1.

2/3

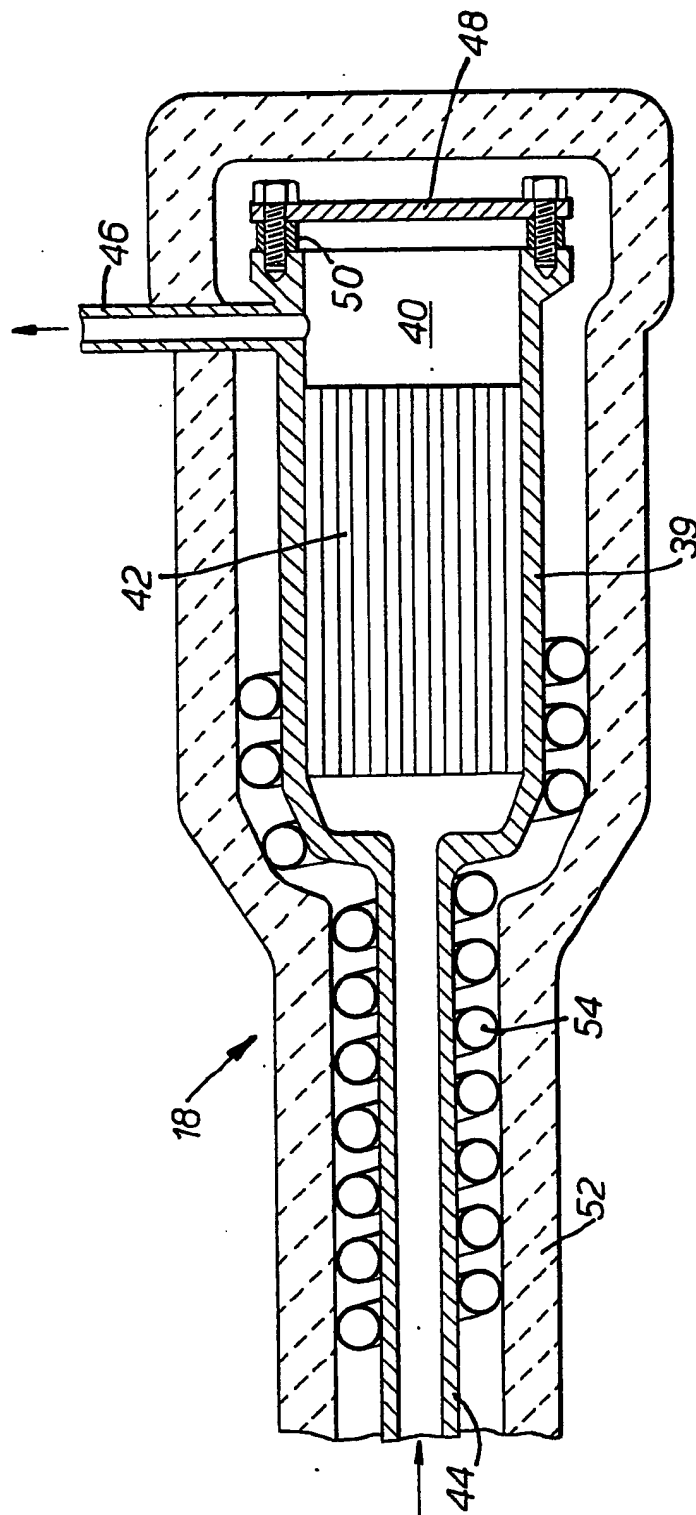


FIG. 2.

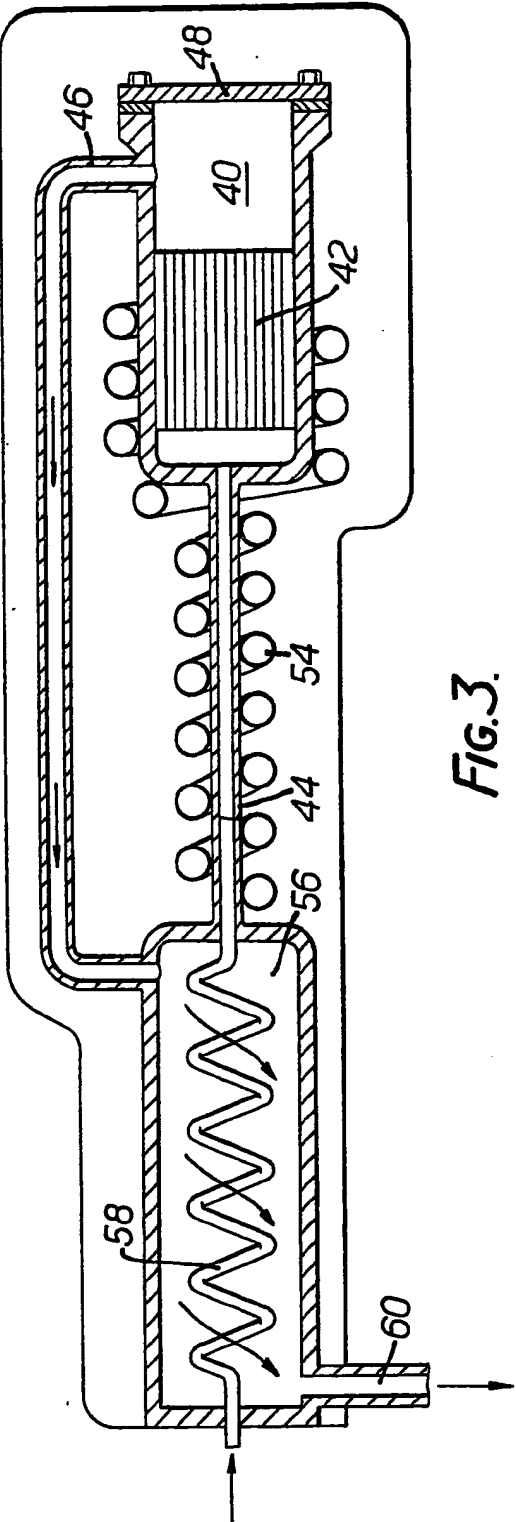


FIG. 3.

SPECIFICATION

Air purification method and apparatus

5 This invention relates to the purification of oxygen-containing gas streams. The invention has particular, although not exclusive, application to the purification of air, and for convenience will hereafter be described in relation to air purification, although the skilled reader will understand that this invention may be used to effect the purification of other oxygen-containing gas streams.

10 For many purposes it is necessary to remove one or more contaminants from an air stream before it can be used. Numerous techniques are employed to purify air streams, amongst which absorption with absorbants such as activated carbon, activated alumina and molecular sieves, and chemical conversion of contaminating substances using such reagents as soda lime are two which are capable of effecting a high degree of contaminant removal.

15 Many air sources contain hydrocarbon impurities, alone or together with carbon monoxide. For example, air which has passed through a high pressure compressor will typically contain about 1.6 ppm of methane and about 5 ppm of higher hydrocarbons, these impurities arising in the main from oxidation of lubricant oil in the compressor at the high temperatures and pressures at which the compressor operates. Even ambient air may often contain hydrocarbon impurities, for example as a result of industrial pollution or even from the presence of background methane in marshy areas. Although seemingly rather small, the concentrations of hydrocarbon or carbon monoxide contamination commonly encountered may in practice cause considerable problems. For some purposes, eg in the medical field or high quality analytical work, the hydrocarbons themselves constitute an impurity which it is desirable if not essential to remove. Even if not themselves a critical impurity in the intended use of the air, the presence of the hydrocarbons and carbon monoxide can deleteriously affect purification agents which are to be employed for the removal of other impurities. For example, activated alumina which has absorbed oil vapours may be impossible to re-activate. Similarly, sodium aluminosilicate molecular sieves, such as the 13X type, will progressively become less efficient absorption agents upon reactivation if used to purify air from a compressor, probably due to the heavier hydrocarbon fractions and oil aerosol present in the air stream becoming deposited on the molecular sieve and then, during reactivation, cracking and consequently blocking the pores of the sieve. Again, soda lime which is used as a reagent for removing carbon dioxide by chemical conversion to sodium and calcium carbonates, may become less efficient due to free oil in the air stream blocking the pores of the material.

20 There is thus a need for a method of purifying a hydrocarbon-contaminated air stream which permits the purification agent used to function efficiently, without being deleteriously affected by hydrocarbon and carbon monoxide contamination. For convenience, the term "hydrocarbon" is used hereinafter

to refer not only to contaminants which are chemically hydrocarbons but also to mixtures of such contaminants and carbon monoxide.

In its broadest aspect, the present invention provides a method for purifying a hydrocarbon-contaminated (as hereinabove defined) air (or other oxygen gas-containing) stream, wherein the gas stream is brought into contact with an oxidation catalyst under conditions which convert hydrocarbon contaminants present therein into carbon dioxide and water, and thereafter the gas stream is brought into contact with one or more chemical purification agents for removal of one or more contaminants present in the gas stream.

70 It is to be understood that any type of, or combination of, chemical purification agent may be used in the second stage of our method, provided only that the purification agent, or combination of agents, removes the substances which have to be removed for the intended end use of the air stream. It will be appreciated that for some purposes it may not be necessary to remove the carbon dioxide and water which are formed during the first stage of the method, although for other purposes these substances will themselves be impurities requiring removal. In all cases, however, the first stage catalytic oxidation of hydrocarbon impurities will result in at least substantially complete removal of hydrocarbon impurities from the air stream, thereby minimising the risk of damaging hydrocarbon contamination of the purification agent or agents subsequently employed. Furthermore, in many instances, hydrocarbons will themselves be impurities from the point of view of the intended end use of the air, and the present invention provides an efficient means for their removal.

80 Any suitable oxidation catalyst may be employed in the first stage of our method, for example a noble metal such as platinum supported on an alumina substrate. However, we have found that best results have been obtained using a noble metal catalyst such as platinum deposited on the steel alloy known as "Fecralloy" in the form of a pliable foil. Such a catalyst is easy to handle yet robust, and enables a large catalytic surface area to be provided within a relatively small volume. The optimum operating temperature for this catalyst is normally in the range of 500°-600°, although it can be used at temperatures down to 350°C or up to in excess of 800°C.

85 Our invention is especially advantageous when applied to the removal of contaminants from compressed air when the compressor is driven from an internal combustion engine. In such a system, the hot exhaust gases from the internal combustion engine may be employed as a source of heat for the catalytic oxidation zone, and also, if necessary, for supplying some of the heat to reactivate the chemical purification agent. Molecular sieves of the 13X type, will be preferred chemical purification agent in many cases because of its ability to efficiently absorb most known contaminants of air, and this absorbing agent is normally regenerated by passing a relatively small flow of air through the absorbant whilst it is heated to about 300°C. By using the hot exhaust gases to supply some of this heat, the power

requirements for the regeneration step may be considerably reduced. Further savings in the power requirement of the system may be achieved by using the heat of the air which has passed through the catalytic oxidation zone to heat the incoming hydrocarbon-contaminated air stream to that zone.

Especially when a re-activatable purifying agent is used in the second stage of the purification method of this invention, it is advantageous to provide two or more separate zones for contacting the air stream with the second stage purifying agent which are arranged in parallel, whereby the air stream can be passed alternately through each zone whilst the purifying agent in the other or another zone is being reactivated. By this means, the system can be operated continuously for long periods.

Apparatus for the purification of a hydrocarbon-contaminated air (or other oxygen gas-containing) stream in accordance with the present invention comprises:

- (a) a catalytic reactor having an air inlet thereto and an air inlet therefrom and defining between said inlet and said outlet a hydrocarbon oxidation zone for contacting an air stream passing through said reactor with an oxidation catalyst for converting hydrocarbon contaminants present in the air stream into carbon dioxide and water;
- (b) means for continuously supplying heat to said hydrocarbon oxidation zone;
- (c) a chamber for containing a heat reactivatable chemical purification agent connected to said outlet from said catalytic reactor for receiving the air stream therefrom and having an outlet for the discharge of purified air which has passed into contact with said purification agent; and
- (d) means for intermittently supplying heat to said chamber to reactivate said chemical purification agent when it has become spent.

Such apparatus preferably includes at least two intermittently heatable chambers for the purification agent which are arranged in parallel, and means for alternately passing the air stream from the catalytic reactor through each chamber in turn whilst heat is supplied to the other or another chamber to reactivate spent purification agent therein.

Advantageously, the catalytic reactor includes heat exchange means for pre-heating the in-flowing air stream by indirect heat exchange contact with the discharging air stream therefrom.

When the apparatus of the invention is to be employed to purify a compressed air stream which has been pressurized by a compressor driven by an internal combustion engine, the means for supplying heat to the hydrocarbon oxidation zone and/or to the chamber or chambers for the chemical purification agent may comprise an indirect heat exchanger receiving the exhaust from said internal combustion engine.

In another aspect, the present invention provides apparatus for the purification of a hydrocarbon-contaminated air stream, which comprises:

- (a) a catalytic reactor having an air inlet thereto and an air outlet therefrom and defining between said inlet and said outlet a hydrocarbon oxidation zone;

- (b) an oxidation catalyst in said hydrocarbon oxidation zone for converting hydrocarbon contaminants present in an air stream passing through said zone into carbon dioxide and water; and

- (c) a chamber containing a chemical purification agent for removing one or more contaminants present in the air stream connected to receive the air stream from the outlet from said catalytic reactor.

The method and apparatus of the present invention are useful for a wide variety of purposes in which purified air is required. By way of example only, there may be mentioned the following applications:

- (i) the provision of a pure air supply for cryogenic devices such as Joule Thomson mini-coolers;
- (ii) for respiratory use such as the charging of sub-aqua bottles or for direct supply to diving or associated equipment;
- (iii) in medical applications requiring pure, sterile air; and
- (iv) in certain analytical procedures eg those requiring the provision of zero gas for gas analyzers or as diluting, blending or purging gases.

The invention will now be described further with reference to the accompanying drawings, in which:

Figure 1 is a schematic diagram illustrating one embodiment of the air purification method and apparatus of the present invention;

Figure 2 is a somewhat schematic cross-sectional view illustrating one form of catalytic reactor useful in the system shown in *Figure 1*; and

Figure 3 is a somewhat schematic cross-sectional view of an alternative form of catalytic reactor useful in the system shown in *Figure 1*.

Referring first to *Figure 1* there is shown a system for purifying a hydrocarbon-contaminated pressurized air stream from an air compressor 10 having an air-intake 12. The pressurized air from the compressor or outlet 14 is led by line 16 to a catalytic reactor 18 which contains an oxidation catalyst for oxidizing to carbon dioxide and water hydrocarbon impurities present in the air stream, for example as a result of its passage through the compressor 10. As schematically illustrated at 20, there is an electric supply for a heating coil (not shown in *Figure 1*) in the reactor 18. A preferred oxidation catalyst is a noble metal such as platinum supported on "Fecralloy", and for such a catalyst the optimum temperature in the oxidation zone of the reactor 18 will typically be about 500°C. Within the reactor 18 the hydrocarbon impurities present in the air stream are oxidized to carbon dioxide and water.

From the reactor 18, the air stream is led by line 22 through a cooler 24, in which water is condensed out of the air stream, and a condensation trap 26 which removes the condensed water, to the second purification stage of the system, generally indicated by reference numeral 28, and in which undesired contaminants in the air are removed by contact with a chemical purification agent which may, for example, be of the absorption or chemical conversion types. Molecular sieve type 13X is a preferred chemical purification agent in many instances.

The second purification stage 28 includes two chambers, 30 and 32, for effecting contact between

the air stream and the chemical purification agent. These chambers are arranged in parallel, with valve-controlled air inlets and outlets, so that the air stream may be alternately passed through each chamber in turn, thereby allowing the chemical purification agent in the chamber through which air is not being passed either to be changed, if it is not reactivatable, or to be reactivated if of a reactivatable type, without interruption of the purification process.

In the embodiment illustrated in Figure 1, heating means shown schematically at 34 are provided for independently heating each chamber 30, 32 during a reactivation cycle. For example, if the chemical purification agent is molecular sieve type 13X, reactivation of spent agent is conveniently affected by heating to 300°C whilst passing a stream of air through the molecular sieve in the opposite direction to that of the air stream during the absorption cycle, ie as shown the purging air is passed upwardly through the chambers. As will be appreciated, the purging air stream can be obtained in the illustrated embodiment by bleeding off a minor proportion of the purified air exiting from the chamber being used for the absorption cycle.

The purified air is removed from the second purification stage 28 by line 36 for, optionally, further purification, or for use. A sampling line 38 is connected to an analyzer (not shown) by means of which the purity of the air product may be continuously or periodically monitored.

One form of catalytic reactor 18 which is suitable for use in the system shown in Figure 1 is illustrated in Figure 2. The reactor includes a generally cylindrical reaction vessel 39 of an oxidation resistant metal, typified by 321 (EN 58 B) stainless steel, which defines a hydrocarbon oxidation zone 40 which contains an oxidation catalyst 42, for example in the form of a roll of "Fecralloy" which supports a noble metal catalyst such as platinum. Air to be purified is admitted into the oxidation zone 40 through an elongate inlet 44 and leaves through outlet 46. Access to the oxidation zone, to insert or replace catalyst, is through a cover 48 which is screwed onto one end of the reaction zone and sealed thereagainst by a seal 50, for example by a nickel-plated copper gasket.

The oxidation zone 40 and the inlet 44 are surrounded by a jacket 52 of refractory insulation material for example silica or zirconia wool which is spaced from the outer wall of the oxidation zone 40. Within the annulus between the insulating jacket and the oxidation zone and air inlet thereto is an electrical heating coil for supplying heat to the oxidation zone.

It will be appreciated that, apart from any heat possessed by the air stream to be purified, all the heat for the oxidation zone of the reactor shown in Figure 2 has to be supplied externally. A modified form of reactor 18 which requires less external heat to be supplied is shown, somewhat schematically, in Figure 3, wherein like reference numerals are used for those parts which correspond to parts in the reactor of Figure 2.

Referring now to Figure 3, the reactor 18 includes an indirect heat exchange section 56 consisting of a

heat exchange coil 58 through which the air to be purified flows before reaching the inlet 44 to the oxidation zone 40, and around which the air which leaves the oxidation zone 40 through outlet 46 flows in heat-exchange contact, as indicated by the arrows, before finally leaving the reactor 18 through outlet 60. By way of example of the savings in the externally supplied heat which may be achieved by the reactor of Figure 3, for a reactor of given volume and air throughput which requires in the Figure 2 arrangement a 1.5 Kw heater 54, a heater of only about 1 Kw would be required in the reactor of Figure 3. Another advantage arising from the provision of the heat exchange section 56 is that it reduces the load on the cooler 24.

Still further savings in external heating requirements may be achieved if the system is to be used to purify air from a compressor. In this situation, hot exhaust gas from the internal combustion engine used to drive the compressor may be employed to indirectly heat the oxidation zone 40 of either the Figure 2 or Figure 3 reactors. For example, a heat exchange coil may be wound around the oxidation zone 40.

Advantage may also be taken of the fact that with some chemical purification agents, for example molecular sieves of the 13X type, the purification reaction is exothermic. This gives rise to the possibility of employing the heat liberated during a, say, absorption cycle to supply the heat needed for the reactivation cycle.

Particularly when the air purification system is under high pressure, care is to be taken to ensure that the apparatus is leak-tight. This is desirable not only from the points of view of safety and minimising loss of purified air, but also, when absorbents are used in the second stage purification, because depressurization of the system will tend to liberate contaminants which had previously been absorbed and which in an unpressurized system may migrate towards the purified air outlets.

Experiments which we have conducted show that the system of Figure 1, using platinum-on-Fecralloy as catalyst in the hydrocarbon oxidation zone and molecular sieve type 13X as the chemical purification agent in the second stage purification, can be operated continuously for up to three months without need to replace either the catalyst or the molecular sieve. Because hydrocarbon impurities are removed from the air before it reaches the molecular sieve, the latter can be reactivated several hundred times without appreciable loss of absorption efficiency. In contrast, the efficiency of the molecular sieve drops to about 25% of its original value after only about eight reactivations if the hydrocarbons have not been first removed from the air. Thus, the present invention can provide a very economical system for purifying air.

Depending of course on the volumes of air or other oxygen gas-containing gases to be purified, the apparatus of the present invention is adapted in many instances to be portable.

CLAIMS

1. A method for purifying a hydrocarbon-contaminated (as hereinbefore defined) air or other oxygen gas-containing stream, wherein the gas stream is brought into contact with an oxidation catalyst under conditions which convert the hydrocarbon contaminants present therein into carbon dioxide and water, and thereafter the gas stream is brought into contact with one or more chemical purification agents for removal of one or more contaminants present in the gas stream.
2. A method according to Claim 1, wherein the oxidation catalyst is a noble metal.
3. A method according to Claim 2, wherein the oxidation catalyst is a noble metal supported on a "Fecralloy" support.
4. A method according to any preceding claim, wherein said chemical purification agent is or comprises a type 13X molecular sieve.
5. Apparatus for the purification of a hydrocarbon-contaminated air or other oxygen gas-containing stream, comprising:
- (i) a catalytic reactor having a gas stream inlet thereto and a gas stream outlet therefrom and defining between said inlet and said outlet a hydrocarbon oxidation zone for contacting a gas stream passing through said reactor with an oxidation catalyst for converting hydrocarbon impurities present in the gas stream into carbon dioxide and water;
- (ii) means for continuously supplying heat to said hydrocarbon oxidation zone;
- (iii) a chamber for containing a heat reactivatable chemical purification agent connected to said outlet from said catalytic reactor for receiving the gas stream therefrom and having an outlet for the discharge of purified gas which has passed in contact with said purification agent; and
- (iv) means for intermittently supplying heat to said chamber to reactivate said chemical purification agent when it has become spent.
6. Apparatus according to Claim 5, including at least two intermittently heatable chambers for the purification agent which are arranged in parallel, and means for alternately passing the gas stream from the catalytic reactor through each chamber in turn whilst heat is supplied to the other or another chamber to reactivate spent purification agent therein.
7. Apparatus according to Claim 5 or Claim 6, including heat exchange means in the catalytic reactor for pre-heating the in-flowing gas stream by indirect heat exchange contact with the discharging gas stream therefrom.
8. Apparatus for the purification of a hydrocarbon-contaminated air or other oxygen gas-containing stream, comprising:
- (i) a catalytic reactor having a gas stream inlet thereto and a gas stream outlet therefrom and defining between said inlet and said outlet a hydrocarbon oxidation zone;
- (ii) an oxidation catalyst in said hydrocarbon oxidation zone for converting hydrocarbon contaminants present in a gas stream passing through said

zone into carbon dioxide and water; and

(iii) a chamber containing a chemical purification agent for removing one or more contaminants present in the gas stream connected to receive the gas stream from the outlet from said catalytic reactor.

Printed for Her Majesty's Stationery Office, by Croydon Printing Company Limited, Croydon, Surrey, 1983.
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.